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Contract No. DA-11-ORD-022-3108

EMBRITTLEMENT OF METALS

BY ORGANIC LIQUIDS

ARF-B183-13
(Quarterly Report)

Commanding Officer
Frankford Arsenal
Philadelphia 37, Pennsylvania

ARMOUR RESEARCH FOUNDATION
of
ILLINOIS INSTITUTE OF TECHNOLOGY
Technology Center
Chicago 16, Illinois

Contract No. DA-11-ORD-022-3108

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December 1, 1962, to February 28, 1963

for

Commanding Officer
Frankford Arsenal
Philadelphia 37, Pennsylvania

Attention: Mr. J. M. McCaughey
Pitman-Dunn Laboratories

March 5, 1963

ARMOUR RESEARCH FOUNDATION OF ILLINOIS INSTITUTE OF TECHNOLOGY

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ABSTRACT

The existing knowledge on the subject in the published literature is reviewed, and a modus operandi is outlined for an extensive study of the incidence of such embrittlement. Evaluation schemes will be based primarily on static and dynamic fatigue tests. A first appraisal of 48 selected organic fluids in static fatigue has shown no propensities for embrittlement.

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I. INTRODUCTION

This report, as well as subsequent ones, summarizes progress in one portion of the larger program entitled "Fracture of Metals," identified under Contract No. DA-11-ORD-022-3108. This report covers the period of December 1, 1962, to February 28, 1963.

Embrittlement of constructional metals by low-melting metals and by certain corrosive solutions and combinations of air, water, and salts are well documented, and a framework of understanding is evolving. The present phase of the larger program sets out to explore the existence and nature of embrittlement which might be produced by exposure to organic liquids and tensile stresses. There is some published evidence of such occurrences but the evidence is fragmentary and often ambiguous. It is considered, therefore, that the first step in the research program is to conduct some simple explorations for embrittlement so that some broad categories can be drawn for organic species which produce this effect. More detailed analysis of mechanism will follow upon a clearer appreciation of the scope and nature of the phenomenon. In this work, organic liquids will be defined as pure liquid species, miscible liquids, and solutions of solids in liquids.

II. INFLUENCE OF ORGANIC AND AQUEOUS ENVIRONMENTS ON THE MECHANICAL PROPERTIES OF METALS--AN APPRAISAL OF THE LITERATURE

In recent years several summaries and interpretations of published works have appeared.[1-4] Each of these has reviewed essentially the same publications, but the interpretations of their significance differ somewhat. In brief review, the following is a list of phenomena revealed by various experiments which are pertinent to the topic under discussion.

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(a) Reduction in the yield point of Sn, Al, and Pb single crystals immersed in surface-active agents such as oleic acid, palmitic acid, and cetyl alcohol as dilute solutions in an inert solvent. These are the work of Rebinder and his associates. The work of Kramer does not confirm the effect on critical resolved shear stress in single crystals of Al but does confirm other effects such as work hardening and creep.

(b) Increase in the rate of creep of single crystals of Sn, Al, Pb, Zn, and Cd. The separate works of Rebinder, Masing, and Kramer are in essential agreement. There are long-standing arguments in the literature based on work by Andrade and by Harper and Cottrell on the presence and significance of oxide films since all of this work has been done on very thin wire shaped specimens.

(c) The creep of polycrystalline wires is unaffected by the presence of organic species of surface-active agents when the grain diameter of the metal is less than about 2% of the wire cross section, i.e., about 50 grains in a section.

(d) A loss of 20-25% in the fracture strength of single crystals of Pb tested in solutions of active fatty acids and alcohols in a neutral solvent at very high strain rates only.

(e) Discontinuous changes in creep rate and flow stress of both single crystal and polycrystalline aluminum subjected intermittently to electrolytic dissolution. The effect is proportional to the electrolytic current or rate of dissolution.

(f) Influence of imposed electrolytic potential on the hardness of Tl, Te, and Cd, which includes both increases and decreases of hardness. The presence of surface-adsorbing alcohols accentuates the effect of potential. The phenomenon is called the Electro Capillary Effect by Rebinder.

(1) E. S. Machlin, Chapter 12 of Strengthening Mechanisms in Solids, ASM, 1962, pp. 375-404.

(2) I. Kramer and L. J. Demer, "Effect of Environment on Mechanical Properties of Metals," in Progress in Materials Science, Vol. 9, No. 3, 1961, 199 pp.

(3) P. Rebinder and V. Lichtman, "Effect of Surface-Active Media on Strains and Rupture in Solids," Proc. Second Intl. Congress of Surface Activity, Butterworth, London, 1957, pp. 563-580.

(4) A.R.C. Westwood, "Effects of Environment on Fracture Behavior," Tech. Rept. No. 62-15 from RIAS, 1962, 105 pp.

(g) Reduction of the order of 25-50% in fatigue strength and endurance limit of ferritic steels immersed in vaseline oil solutions containing isoamyl alcohol or oleic acid. Simple high-purity water can produce the same deterioration by what is termed "corrosion-fatigue." The addition of isoamyl alcohol to water reduces the rate of corrosion yet perpetuates the fatigue strength loss and seems to be a distinguishable and separable effect.

There are various proposed mechanisms for these effects. The Rebinder group prefer the concept that physical adsorption of polar molecules of certain species lowers the surface energy of the solid leading to two effects-- reduction of the magnitude of barriers to slip at the surface leading to accelerated yielding and reduction of the cleavage energy at the surface leading to cracking. Simple chemical dissolution or electrolytic polishing can, in principle, lower the flow stress or raise it depending on the nature of the surface material. If the surface constitutes an oxide film and/or structurally irregular material, the surface dissolution will naturally lead to a reduced flow stress but generally only in fine wire or foil where the crystal size is not much smaller than the thickness or diameter dimension. In brittle materials where cleaved surfaces have a high population of slip sources, the removal of these by chemical dissolution can clearly lead to magnified strength.

Recent experiments by Kramer[5] on Al alloys specimens of large dimensions compared to their grain size suggest that simple material removal models are inadequate, for it was shown that certain reversible yield stress drops occurred with the imposition of electrolytic current. The effects were very small simply because a change in the flow stress of the surface material can have only a small effect on the average flow stress of the specimen. These results seem to be related to the Rebinder electro-capillary effect.

There is an intermediate effect involving chemical attack of the metal by the liquid environment which produces a metallo-organic species, which in turn has the character of a surface-active agent. Kramer's studies on metal stearates and stearic acid and other organic acids justify a process of change of mechanical properties by chemi-adsorption.

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[5] I. R. Kramer, "Effects of Surface Environments on the Mechanical Behavior of Metals," ASD-TDR-62-99 (1962).

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The influence of environment on the plastic flow properties of metals has questionable relevance to present interests in the fracture of normally ductile metals. The most interesting experiments in this field are those by Karpenko on the dynamic fatigue strength of steel in surface-active liquid environments. It is useful at this point to recapitulate his results.

steels: 0.15-0.25% C, 0.7-1.0% Cr
0.35-0.45% C, 0.5-0.8% Mn, 0.8-1.1% Cr.

condition: quenched and tempered, undesignated strength
annealed, UTS = 70,000 psi.

media: air, water, water + 2% isoamyl alcohol,
inactive vaseline oil + 2% isoamyl alcohol
or 2% oleic acid, commercial lubricating
oil, new and spent, lubricating oil + 2%
oleic acid.

Compared to the endurance limit in air, the endurance limits in water, water + isoamyl alcohol, vaseline oil + isoamyl alcohol or oleic acid, and lubricating oil + oleic acid are measurably lower. Water is as effective as any of the surface-active agents by generating what is known as corrosion fatigue. The corrosion process can be stopped at least by visual inspection by the addition of small amounts of isoamyl alcohol as an inhibitor. The surface-active character of isoamyl alcohol is sufficient to produce just as severe loss in endurance limit. There is a distinction between corrosion fatigue and surface-active agent fatigue. In the former case there is no defined endurance limit, whereas in the latter a clearly resolvable limit is measured. This signifies that fracture in the presence of surfactants (a term commonly used for surface-active agents) is dependent on the conjoint existence of a minimum stress and a minimum activity. In corrosion fatigue no critical stress limit exists.

The amount of isoamyl alcohol in water is not critical between 0.2 and 2% concentration. The loss in endurance limit in annealed steels is only of the order of 10%. At an intermediate hardness level, the loss increases to about 20%, and at higher hardness levels (probably UTS = 150,000 psi) the loss in endurance limit is nearly 50%. Thus, the harder the steel, the more prone it is to embrittlement. This is in agreement with all other environmental embrittlement phenomena. Certain commercial

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oils have mild surfactant properties with consequent loss in endurance limit. There was no observed differences between new and spent oil. In the presence of a strong surfactant, specimen finish becomes an unimportant factor whereas it is definitely so in normal testing.

The rate of cycling in fatigue testing is an important factor in life-time in the presence of a surfactant. The lower the rate of cycling, the more effective is the embrittlement. For example, water + 2% isoamyl alcohol lowered the endurance limit by 7% at 10,000 cpm and by 30% at 3000 cpm. This is an important observation for it indicates that the process is controlled by the rate at which the surfactant can move in cracks. Strangely, although the work of Karpenko is at least ten years old, it has never been repeated.

III. <u>CONCEPTS OF EMBRITTLEMENT</u>

The contemporary theories on mechanism of brittle fracture can be described either in terms of balance of forces or in terms of balance of energies. We start from the same base line that, by the application of sufficient stress, slip bands in the individual crystals of a solid aggregate are activated and forced to propagate until obstructed by some barrier which may be a grain (intercrystal) boundary or an array of hard particles. The obstructed slip band generates a magnified stress field in the immediate vicinity of the point of obstruction. The intensity of the shear and normal stress components of this local stress field is proportional to the applied stress and to the length of the slip band. When the local stress reaches the critical stress for cleavage of the material involved, a microcrack will be generated. Usually the stress sufficient to initiate microcrack is also sufficient to propagate it. If the critical shear stress to penetrate the barrier by slip is reached before the critical cleavage stress, slip will propagate past the barrier point, and the magnified stress field will relax. This is why low-strength metals are generally less brittle than high-strength materials.

The influence of environment is upon those obstructed slip bands which develop at the surface of the solid metal. We conceive that liquids which wet the metal surface provide a reservoir of highly mobile atoms or molecules which can approach and interact with the electronic fields of the

surface metal atoms. Certain species of atoms or molecules should, in principle, interact with the surface metal atoms to reduce their cohesive strength, which is essentially the cleavage strength. Thus a system of forces develops whereby external force generates local stress intensifica-tion, which at fracture equates to the intrinsic cohesive or cleavage strength of the metal. This, in turn, may be significantly reduced by surface adsorp-tion of certain atom or molecule species. The same argument can be re-stated in terms of the equation of local regions of high strain energy equating to a critical expenditure to form new surface and the surface energy requisite being reduced by the presence of surface active agents.

The bond strengths in a metallic solid are quite high compared to nonmetallic solids of the long chain or large molecule construction. It is therefore not surprising that low-melting liquid metals (the melting point is not a criterion of bond strength) can be very severe embrittling environments. The bond strengths in organic liquids are much weaker even at the level of molecule dissociation so that we cannot expect as strong interaction with a metallically bonded structure. However, a large family of metallo-organic compounds exists which ought to provide an intermediate effectiveness if the electronic field in the metallo-organic molecule can be made to interact with a metallic lattice.

IV. EMBRITTLEMENT OF METALS BY ORGANIC
LIQUIDS--EXPERIMENTAL METHODS

In a subject area as virgin as this, the number of combinations of metals, alloys, metallurgical conditions, stress conditions, manner of test-ing and environment candidates is colossal. We must therefore select those combinations of circumstances which by logic and prior experience are most likely to produce premature fracture. By analogy with experience in liquid-metal and stress-corrosion cracking, some very specific simplifications can be chosen. We shall confine our work to two engineering metals--Al and Fe--which offer the greatest potential spectrum of strengths and structures. We choose for each an alloy capable either by aging or by quench and temper to produce high strengths by dispersion of fine particles in the structure. In the case of Al the alloy 2024 has been chosen, heat-treated to the T3 con-dition where it possesses a yield strength of 47,000 psi. The Fe counterpart
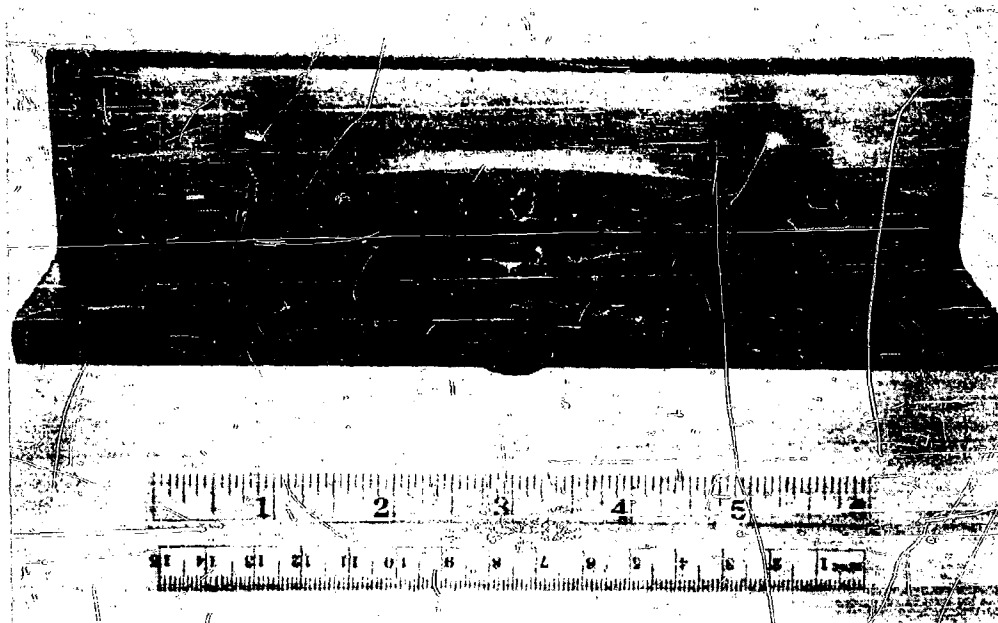
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is an air hardenable, high-strength steel (commercial designation 300 M[*]) quenched and tempered to a yield strength of 204,000 psi. All previous experiences point to these conditions as most likely to exhibit embrittlement.

There are three test systems which seem likely to exhibit some type of measurable embrittlement. These must be discussed individually. The common problem is to achieve actual wetting of the metal by the liquid. Steel and aluminum, no matter what the means of surface preparation, possess a complete oxide film covering. Most organic fluids wet the oxide film as easily as the metal; but in terms of the mechanisms discussed, this could be a completely innocuous situation. A direct method of oxide removal is by the use of corrosive acids or alkalis even in dilute form. There are limitations to this approach. Most of the interesting organic candidates are not miscible or soluble in aqueous solutions, and conversely the acids and alkalis are not soluble in organic solvents. With steel, acid attack involves the generation of hydrogen, which is readily adsorbed into the steel. Hydrogen is capable of producing embrittlement of the same nature and degree in high-strength steel under stress. It is therefore very important to design experiments such that hydrogen embrittlement is either excluded or separately distinguishable.

The first test system sets out to produce delayed failure in statically loaded specimens. Both of these materials loaded in tension up to the tensile strength will sustain these stresses indefinitely. Brittle failure after a limited period of time is characteristic of environmental embrittlement and hydrogen cracking (which might be regarded as an internal environment). The delayed failure test has virtue in its simplicity of operation and interpretation. Figure 1 illustrates a bend fixture designed for this research program. The specimen is a strip-shaped coupon of dimensions 5 in. long x 0.5 in. wide x ~ 0.080 in. thick. The specimens in the heat-treated condition are ground on the tension surface. Each specimen is loaded to its yield point in the bend fixture by means of an Instron universal testing machine. The load is transferred from the machine to the center pin by means of a threaded nut. The center loading pin transmits load to the specimen by a solid glass cylinder, which effectively provides line contact and electrical insulation. Load is

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[*] 0.40% C, 1-6% Si, 0.75% Mn, 0.85% Cr, 1.85% Ni, 0.40% Mo, 0.08% V.

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Figure 1

PHOTOGRAPH OF STRESSED SPECIMEN SET IN STATIC FATIGUE
BEND FIXTURE. The two end pins are electrically separated from the
specimen by insulating tape. The center loading contact with the speci-
men is by means of a glass cylinder. The specimen is thus isolated
from spurious electrolytic potentials. In stressing, the center screw
is actuated by the platen of a precision universal testing machine.
When the correct stress has been applied to the specimen, the load is
transferred from the machine to the nut on the screw shown.

transferred from the machine to the loading pin by an intervening steel ball. The steel ball and glass cylinder jointly ensure good axiality of loading. The two outside support pins are covered with insulating tape so that no electro-chemical currents can be generated between the specimen and the loading fixture.

A drop of the candidate fluid is placed on the ground surface of the tension side. With a carbide tool, the ground surface is scraped in a direction transverse to the grinding marks. The tool point is essentially sub-merged below the candidate fluid to encourage true wetting with the metal. A reservoir of fluid is provided by clamping a cotton wad saturated with fluid to the tension surface on top of the scribe marks. The whole assembly is placed inside a glass bottle and sealed off by means of a rubber-gasketed steel cap. This is necessary because of the volatility of some of the fluid candidates. It will also be possible to conduct this preparation and encapsulat-ing in an argon chamber for those instances where the candidate is reactive with air. About 100 of these test fixtures have been manufactured in order to permit simultaneous exploration of a large number of candidates for both steel and aluminum. Each test is run for a week with daily checks for inci-dence of failure.

A second approach to embrittlement evaluation involves tests simi-lar to those of Karpenko. It is planned to evaluate premature failure in dy-namic fatigue. This has advantage in that a crack must form and propagate so that there is no question of continuous contact between the candidate liquid and fresh metal surface. The test approach will be somewhat different from that used by Karpenko. There is no real need to generate a full stress failure cycle (S-N) curve. It is sufficient to choose a stress which will produce systematic failure in air in an arbitrary number of cycles--say 500,000--and compare this to the number of cycles to failure when the crack zone is immersed in the candidate fluid. A cycling rate of 1800 per minute should be quite appropriate from the published results of Karpenko. A sheet type specimen with two edge notches along an axis transverse to the applied stress will be used. In order to prevent undesirable specimen flexure, the tests will be conducted in pull-pull--i.e., from a low tensile stress to a high tensile stress. The same materials for test(Al alloy and 300 M steel)will be used.

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A third approach to embrittlement possibilities is worth some preliminary exploration. The electro capillary effect in conjunction with solutions containing surface-active media has not been properly explored. It is planned to conduct a series of experiments with the Al alloy using an aqueous solution which can remove the oxide but will not produce stress-corrosion cracking. The tensile behavior -yield, ultimate strength, and ductility -can be studied as a function of the species of surface-active agent and the potential between the test specimen and an inert electrode.

V. CONSIDERATIONS IN THE SELECTION OF ORGANIC COMPOUNDS

To determine whether organic compounds can cause gross losses in the integrity of structural metals such as steel and aluminum, the selection of organic compounds for trial is necessarily a matter of judgment based on past research on metal embrittlement. Such judgment attempts to select those materials which are deemed most likely to cause failure because of their structural chemistry and physical properties and to examine these under conditions which maximize such effects.

From analogy with factors involved in established deteriorative processes, the molecular species under consideration must be mobile and must be adsorbed in some manner on the clean metal surface. It most pro-duce an oriented layer on the metal surface with an adequate reservoir of immediately available molecules to extend this oriented layer rapidly onto new metal surfaces displayed by flaws or cracks created under physical stress.

To meet these requirements, the organic material must be used as a mobile fluid -that is, as a gas or as a liquid or solution of relatively low viscosity. To be adsorbed, the molecule must possess at least one polar center which has a strong attraction for the clean metal surface. It is likely that the force of such metal surface attraction or adsorption must be high to provide at least part of the energy required to overcome surface cohesive forces in the metal. In metal embrittlement, the molecules must cover the walls of an incipient crack or flaw and follow the crack as rapidly as it grows. The ideal adsorbed layer would be expected to lower the external

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stress required to cause the crack to continue through the metal mass. It is to be noted, however, that the actual available energy from the adsorption process is reduced by a quantity related to the interattractive forces between the organic molecules themselves. Other things being equal, such interattractions are least in gases and liquids; organic molecules with this property are most commonly found in the smallest and simplest members of a homologous series.

To some extent at least, molecular size would also be expected to represent a limiting factor in the ability of an organic molecule to enter a submicroscopic flaw or crack and to penetrate to its tip. However, long thin molecules could accomplish this with perhaps the same facility as do shorter molecules of the same type; the adsorbed layer would be less crowded, but the effect might be the same.

Hence, the organic material must be fluid and sufficiently polar to be strongly attracted to the metal. There is nothing to say, however, that a paste made of a solid organic material and an inert liquid will not work, even though the solid is sparingly soluble in the liquid. If the active material is adsorbed at the surface of the metal as the first step in the mechanism and if the liquid phase is maintained at saturation by an available reservoir of undissolved solid, conditions for embrittlement might be readily met. One must recognize that if adsorption is essential, maximum adsorption can be achieved at least as readily by using a poor solvent as by using a good solvent for the same material. Adsorption from solution represents a competitive partition between the surface and the liquid phase. A strong affinity by the metal surface for the compound can be offset by an even stronger affinity by the solvent; the better the solvent, the stronger the affinity. This says that, other things being equal, it is best to use a solvent which dissolves only enough material to provide an ample supply of solute for action on the surface.

The metal organics to be studied will emphasize compounds containing metals found to be effective in the elemental liquid state as agents for embrittlement of steel or aluminum. The metal organics will include alkyl and aryl derivatives, chelates, and salts of organic acids. Metal hydride selection is very limited; most of these are high-melting, insoluble solids.

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Metal chelating agents will also be studied particularly those which form chelates with aluminum or iron. Acetylacetone, EDTA, and similar sequestering agents are representatives of this class. Metal salts of long-chain organic acids are of particular interest because of their strong adsorptive properties.

Examination of commercially available surface-active agents is prompted primarily by reports of degradation caused by substances which contain such agents (i.e., red ink).

Several compounds are of interest because of their small molecular size and strong polarity; these would include hydrazine, acetylene, formaldehyde, phosgene, and several others.

Surface-active agents in the sense used by Rebinder merit close examination because of their reported effects on strength and corrosion fatigue. These compounds include long-chain fatty acids, alcohols, and primary amines. All are strongly adsorbed on clean metal surfaces.

These seemingly restricted classes represent a fairly formidable array of candidates. The materials to be tested are currently selected on the basis of ... ability to experimental conditions. However, it is expected that experimental procedures will be changed when necessary to accommodate volatile materials and those which are abnormally sensitive to moisture and air.

VI. <u>EXPERIMENTAL RESULTS</u>

A large number of static fatigue tests have been performed involving both the Al alloy and the hardened steel, stresses at the yield point, exposure times of one week encapsulated in glass bottles, and 48 candidate liquids. No failures have been encountered in any of these. The following is a list of the organic candidates which thus far have produced no failures:

    n Hexylamine
    n-Butanol
    Dibutyl carbitol
    2-Ethylhexanoic acid
    n-Butyric acid
    Light white mineral oil
    2-Ethylhexanol

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n-Butylamine
Oleic acid
n-Butanol- 50% solution in dibutyl carbitol
n-Hexylamine -50% solution in dibutyl carbitol
n-Butylamine-50% solution in dibutyl carbitol
Ethoxylated stearylamine
Recinoleic acid (castor oil)
Stearic acid · 1.5% solution in dibutyl carbitol
Cetyl alcohol -1.8% solution in para... n o'l
Stearic acid--saturated solution in paraffin oil
2,4,6-Trimethylbenzoic acid--1% solution in dibutyl carbitol
2,4,5-Triethoxybenzoic acid--2% solution in dibutyl carbitol
Halocarbon oil
Dibenzylamine
Benzyl alcohol
2,4,6-Trinitrobenzoic acid--1.3% solution in dioctyl phthalate
Triphenylacetic acid--1.5% slurry in halocarbon oil
m-Toluidine-50% solution in dioctyl phthalate
o-Chloroaniline-50% solution in dioctyl phthalate
p-Nitroanaline--7.5% solution in dioctyl phthalate
Phenylhydrazine
Dimethylformamide 33% solution in halocarbon oil
Dioctyl phthalate
2,4-Dinitrophenylhydrazine -1.6% solution (saturated)
   in dioctyl phthalate
o-dihydroxybenzene-- 3.3% solution in dioctyl phthalate
o-methylphenol-50% solution in dioctyl phthalate
o-nitrophenol-10% solution in dioctyl phthalate
N,N-dimethyl-o-toluidine 10% emulsion in heavy mineral oil
Bromoacetic acid 10% solution in dioctyl phthalate
Crotonic acid--7% solution in dioctyl phthalate
Hydroquinone -2.2% solution in dioctyl phthalate
Decane phosphoric acid--2.1% suspension in dioctyl phthalate
Lauric acid (coconut oil)- 6.6% solution in heavy mineral oil
Diphenyl sulfone -1.5% suspension in heavy mineral oil
Cetyldimethylbenzyl ammonium chloride--4.7% solution in
   heavy mineral oil
Benzyltrimethyl ammonium chloride--61% solution in water
Dilauryl sulfone -2.2% suspension in heavy mineral oil
Dimethyl sulfate
Hexachlorodiphenyl oxide--1.1% solution in heavy mineral oil
Urea--0.5% solution in dioctyl phthalate
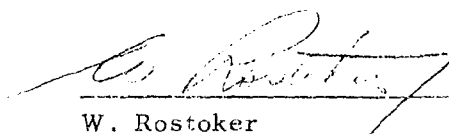Oxalic acid-1% solution in heavy mineral oil.

VII. PERSONNEL AND LOGBOOKS

The research program is under the supervision of the writer with technical consultation by Mr. R. H. Crouse, Senior Chemist. The work it self is being performed by Mr. H. Nichols, Associate Metallurgist, and Mr. R. Sarocco Technician. The results reported are contained in ARF Logbook No. C 13011.

Respectfully submitted,

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W. Rostoker

Tech Rev: JFR

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